REMARKS

This is in response to the Office Action that was mailed on August 27, 2003. Claim 1 is amended to recite the recitations of claims 2 and 7. Claims 2 and 7 are cancelled, without prejudice. A minor formal amendment is made to claim 11, and as amended, claim 11 is recast in independent form. No new matter is introduced by this Amendment. Entry of this Amendment in order to place the application into condition for allowance, or into better condition for appeal, is respectfully solicited. With this Amendment, claims 1, 3-6, and 8-11 are in the application.

Claims 1-11 were rejected under the second paragraph of 35 U.S.C. §112 as failing to define the invention properly. The Examiner argues that it is unclear exactly what the pressure category PN represents with respect to the pipe, other than it is something measured by ISO standard 4065. In fact, the PN category describes the relationship between the nominal wall thickness and nominal outside diameter of the pipe, also taking into account the material properties. Enclosed is a copy of ISO standard 4065. According to ISO standard 4065, thermoplastic pipes subjected to internal pressure are commonly defined as pressure categories PN, which are elaborated in ISO standard 4065. Also enclosed is a copy of pages 65-67 of "Plastic Pipes for Water Supply and Sewage Disposal" by Lars-Erik Janson, 1989. This publication indicates that these nominal pressures – that is, PN – correspond to the respective maximum

permissible working pressures, as expressed in bars. Further, pages 65-67 of the Janson publication describe the relationship between ISO standard 4065 and PN values. The use of this ISO standard and the corresponding PN categories are well within the knowledge of those skilled in the art of pipe technology. It is respectfully submitted, therefore, that the claims herein satisfy the requirements of the statute.

Claims 1, 4, and 8-11 were rejected under 35 U.S.C. §103(a) as being unpatentable over US 5,236,018 (Kobayashi). Claims 2 and 3 were rejected under 35 U.S.C. §103(a) as being unpatentable over Kobayashi in view of US 5,842,505 (Tokui). Claims 5-7 were rejected under 35 U.S.C. §103(a) as being unpatentable over Kobayashi in view of DE 25 51 525 (Vegt). Inasmuch as this Amendment incorporates recitations of both claim 2 and claim 7 into independent claim 1, no claim equivalent in scope to any rejected claim is now pending, and none of the rejections of record is applicable to the claims as amended herein.

Nevertheless, Applicants wish to make the following observations:

The differences between the pipes disclosed by Kobayashi and the pipes according to the present invention result from their totally different preparation methods. Kobayashi clearly discloses pipes ***formed by laminating*** a plurality of fiber-reinforced plastic layers. Laminating/winding is a completely different technique from cone extrusion, and the resulting pipes are significantly different,

since with cone extrusion the whole bulk of the pipe is oriented. This means that the entirety of the matrices are oriented, as can be seen from the last paragraph on page 6 of the present specification and from claim 11 herein. Moreover, as the Examiner has acknowledged, Kobayashi does not disclose the melt flow rate of the polyolefin resin.

Tokui discloses a resin with an MFR between 0.05-20 g/min, measured according to ASTM 1238, condition E, column 2, line 61. This ASTM standard does not disclose what is meant with condition E, that is, what temperature and load are used. The corresponding ISO standard 1133 (copy enclosed), which is referred to in said ASTM standard, clearly states that in E the temperature is 190°C and the load is 0.325 kg. Thus, the MFR of Tokui and the MFR of the present invention are not at all comparable. Based on this, combining the disclosure of Tokui with that of Kobayashi would not direct a person of ordinary skill in the art to the present invention.

Vegt discloses only fiber-reinforced pipes, and is silent about pressure pipes and the cross-orientation of fibers in the pipes. Orientation in angle does not mean the same thing as cross-orientation. Vegt discloses fibers having fiber lengths of 0.2-5 mm, teaching that only short fibers should be used. In the present invention, the length of fibers is 2-15 mm. Moreover, the amount of fibers in Vegt is at most 45%, whereas in the present invention the amount can range up to 75%. Vegt suggests to those of ordinary skill in the art that only

minor amounts of short fibers should be used, which clearly teaches away from the present invention.

It is submitted for the reasons stated above that the present claims define patentable subject matter. In any case, as noted above, inasmuch as this Amendment incorporates recitations of both claim 2 and claim 7 into independent claim 1, no claim equivalent in scope to any rejected claim is now pending, and none of the rejections of record is applicable to the claims as amended herein. Accordingly, it is respectfully submitted that this application has been placed into condition for allowance.

If any questions remain regarding the above matters, please contact Applicants' representative Richard Gallagher (Reg. No. 28,781) at (703) 205-8008.

If necessary, the Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit

Account No. 02-2448 for any additional fees required under 37 C.F.R. § 1.16 or under 37 C.F.R. § 1.17; particularly, extension of time fees.

Respectfully submitted,

BIRCH, STEWART, KOLASCH & BIRCH, LLP

By Richard Gallagher #28,781

for Andrew D. Meikle
Reg. No. 32,868

P. O. Box 747
Falls Church, VA 22040-0747
(703) 205-8000

ADM/RG RG

Thermoplastic pipes – Universal wall thickness table

1 SCOPE AND FIELD OF APPLICATION

This International Standard sets out a universal wall thickness table for thermoplastic pipes.

2 REFERENCES

ISO 161, *Thermoplastics pipes for the transport of fluids – Nominal outside diameters and nominal pressures.*

ISO 497, *Guide to the choice of series of preferred numbers and of series containing more rounded values of preferred numbers.*

3 BASIC THEORY

Generally speaking, the wall thickness of thermoplastic pipes can be expressed as follows :

$$e = f\,(d_e; A; B; C; D) \qquad \ldots (1)$$

where

e is the nominal wall thickness;

d_e is the nominal outside diameter;

A represents the physical influences (temperature, time);

B represents the mechanical influences (internal pressure, external forces);

C represents the chemical influences (contact reactions);

D represents the material properties (long-term behaviour, temperature dependence, chemical resistance).

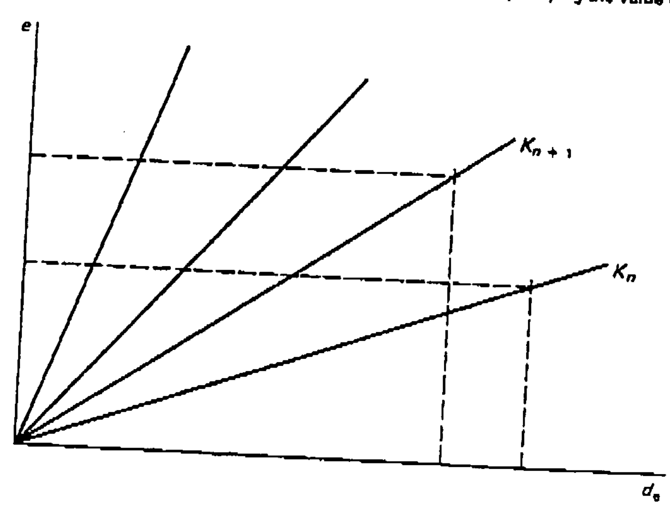
The simplest and (as will be shown later), for standardization purposes, a particularly apt version of equation (1) is :

$$e = K \times d_e \qquad \ldots (2)$$

where K represents all application and material dependences A, B, C and D mentioned in equation (1).

However, equation (2) can also be intrepreted as a combination of purely geometrical values (e; d_e), K being a parameter.

As the graph below shows, any combination of e and d_e can be obtained by varying the value of K.



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From the point of view of standardization, this fact implies the necessity of finding a minimum number of values for K covering all pipe applications. Regarding these applications, the multitude of subjects under discussion in the working groups of TC 138 may be considered as representative. These subjects can be subdivided into the following two main groups :

a) *Pipes predominantly subject to internal pressure*

1) Pipes for the transport of cold water (pipes for water supply).

2) Pipes for the transport of water at elevated temperatures (hot water installations).

3) Pipes for the transport of fluids other than water (pipes for the chemical industry; pipes for the transport of gaseous fuels, except those operating at working pressures which are inadequate to overcome the influence of the external load).

b) *Pipes not subject to internal pressure*

This group comprises especially soil and waste pipes above ground as well as drainage and sewer pipes under earth load, transporting waste water or other fluids by gravity, not only at temperatures up to 20 °C but also at elevated temperatures.

4 PIPES PREDOMINANTLY SUBJECT TO INTERNAL PRESSURE

According to ISO 161, the wall thickness formula

$$e = \frac{1}{(2\,\sigma/p) + 1} \times d_e \qquad \ldots (3)$$

where

σ is the induced stress, and

p is the pressure of the fluid,

applies originally only to subgroup a) 1) as defined in clause 3.

Equation (3) can also be considered as valid for subgroups a) 2) and a) 3) if the value of σ is chosen appropriate to the particular application. In this case, σ and p comprise all application and material dependences A, B, C and D mentioned in equation (1). By expressing these dependences merely as a summary value $\sigma/p = S$, equation (3) may be transformed into :

$$e = \frac{1}{(2\,S) + 1} \times d_e \qquad \ldots (4)$$

An identity between equations (2) and (4) requires that all values of K can be converted into corresponding values of S in agreement with the equation

$$K = \frac{1}{(2\,S) + 1} \qquad \ldots (5)$$

This knowledge facilitates the selection of values of K for group a), namely :

As already mentioned, in the case of group a), S has the meaning of σ/p. In accordance with ISO 161, the values of p have to be preferred numbers of the R 10 series for determining wall thicknesses of thermoplastic pipes. The values of σ used in the past for calculation are fortunately also numbers of the R 10 series. Hence S is always the quotient of two numbers of the R 10 series and therefore in principle is itself a number of the R 10 series. This is the key to the reduction of the numerous combinations of σ and p to a small selection of values of S.

Since preferred numbers are rounded off from theoretical values (calculated values : see ISO 497), quotients of preferred numbers cannot basically be identical either with preferred numbers or with their theoretical values. The latter, however, may be considered as mean values of all corresponding quotients. Therefore, a universal wall thickness table mathematically built up on the theoretical R 10 values for S guarantees a minimum of deviations from wall thicknesses already laid down in existing standards and in papers of working groups.

The table on page 4 is the result of calculations based on the above procedure.

The nominal wall thicknesses (e) are expressed in millimetres to one decimal place and rounded up to the nearest 0,1 mm, if the second decimal is higher than zero.

5 PIPES NOT SUBJECT TO INTERNAL PRESSURE

The numerous pipe applications to be classified into group b), according to clause 3, are characterized only partially by a strictly linear relationship between e and d_e for pipes of identical material and similar conditions of use. Group b), therefore, does not represent the same justifying background for equation (2) as does group a). Yet, in many cases at least an approximately linear relationship between e and d_e is given. Moreover, the available basis for determining dimensions is very often much too rudimentary to permit convincing motivation for the creation of specific pipe series.

These facts justify the hope that an analogous method of selecting values for K as developed for group a) might also be appropriate for group b). This assumption is actually true.

In consequence, there is no reason why pipes of group b) should be classified differently from those of group a).

6 CONCLUDING REMARKS

The practice of selecting minimum wall thicknesses higher than the theoretical values (if technical reasons are given), as well as the possibility of switching over, within the same application, to other series of the universal table, guarantee that the latter will provide a satisfactory solution to any future demand. Neither are complications to be expected

from new materials for pipes, owing to the fact that in view of values of σ to be used for determining dimensions of pressure pipes, the R 10 series of preferred numbers offers a sufficiently differentiated selection to meet all technical and economic aspects. The assumption that a closer graduation in the σ-selection could lead to economical advantages is an error. The ratio $\left(\sqrt[10]{10} \approx 1{,}26\right)$ characterizing the R 10 series is adequate in view of the known scattering results of long-time tests as well as of the problems raised by their extrapolation and the determination of suitable factors of safety. Hence, a closer graduation of the σ-selection would be beyond the possibility of a serious differentiation.

For those exceptions where the general rule is not applicable for technical reasons, it may be necessary to take into account limited variations of wall thickness when preparing documents for these specific applications.

The advantage of a designation of series by means of K or its reciprocal value lies in the additional information concerning the relationship d_e/e.

The mentioned advantage is also given for the designation of series with S being in accordance with equation (4) :

$$S = \frac{1}{2}\left(\frac{d_e}{e} - 1\right) \approx \frac{1}{2} \times \frac{d_e}{e} \qquad \ldots (6)$$

Thus, any pipe can easily be classified on the basis of d_e and e.

In addition, the S-values have the specific advantage of not forming a number series by chance but a pure series R 10. Furthermore, S has in the case of pipes belonging to group a) the meaning of σ/p. That involves advantages for the dimensioning of hot water installations, and industrial pipes in particular. In consequence, there is no doubt that the designation of pipe series as used in the following table represents the optimal solution.

It will be of interest to note that the principles contained in this document have been used for a number of years in the United States of America in the form of the Standard Dimension Ratio (SDR) series. The relationship between the "SDR" and the "S", as defined in this document is given by the equation :

$$\mathrm{SDR} = \frac{d_e}{e} = 2\,S + 1 \qquad \ldots (7)$$

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TABLE – Nominal wall thicknesses (e), in millimetres, of the pipes series

Nominal outside diameter d_e mm	Pipe series S														
	2,5	3,2	4	5	6,3	8	10	12,5	16	20	25	32	40	50	63
2,5	0,5														
3	0,6	0,5													
4	0,7	0,6	0,5												
5	0,9	0,7	0,6	0,6											
6	1,0	0,9	0,7	0,6	0,5										
8	1,4	1,1	0,9	0,8	0,6	0,5									
10	1,7	1,4	1,2	1,0	0,8	0,6	0,5								
12	2,0	1,7	1,4	1,1	0,9	0,8	0,6	0,5							
16	2,7	2,2	1,8	1,5	1,2	1,0	0,8	0,7	0,5						
20	3,4	2,8	2,3	1,9	1,5	1,2	1,0	0,8	0,7	0,5					
25	4,2	3,6	2,8	2,3	1,9	1,5	1,2	1,0	0,8	0,7	0,5				
32	5,4	4,4	3,6	2,9	2,4	1,9	1,6	1,3	1,0	0,8	0,7	0,5			
40	6,7	5,5	4,5	3,7	3,0	2,4	1,9	1,6	1,3	1,0	0,8	0,7	0,5		
50	8,3	6,9	5,6	4,6	3,7	3,0	2,4	2,0	1,6	1,3	1,0	0,8	0,7	0,5	
63	10,5	8,6	7,1	5,8	4,7	3,8	3,0	2,4	2,0	1,6	1,3	1,0	0,8	0,7	0,5
75	12,5	10,3	8,4	6,8	5,5	4,5	3,6	2,9	2,3	1,9	1,5	1,2	1,0	0,8	0,6
90	15,0	12,3	10,1	8,2	6,6	5,4	4,3	3,5	2,8	2,2	1,8	1,4	1,2	0,9	0,8
110	18,3	15,1	12,3	10,0	8,1	6,6	5,3	4,2	3,4	2,7	2,2	1,8	1,4	1,1	0,9
125	20,8	17,1	14,0	11,4	9,2	7,4	6,0	4,8	3,9	3,1	2,5	2,0	1,6	1,3	1,0
140	23,3	19,2	15,7	12,7	10,3	8,3	6,7	5,4	4,3	3,5	2,8	2,2	1,8	1,4	1,1
160	26,6	21,9	17,9	14,6	11,8	9,5	7,7	6,2	4,9	4,0	3,2	2,5	2,0	1,6	1,3
180	29,9	24,6	20,1	16,4	13,3	10,7	8,6	6,9	5,5	4,4	3,6	2,8	2,3	1,8	1,5
200		27,3	22,4	18,2	14,7	11,9	9,6	7,7	6,2	4,9	3,9	3,2	2,5	2,0	1,6
225			25,1	20,5	16,6	13,4	10,8	8,6	6,9	5,5	4,4	3,5	2,8	2,3	1,8
250			27,9	22,7	18,4	14,8	11,9	9,6	7,7	6,2	4,9	3,9	3,1	2,5	2,0
280				25,4	20,6	16,6	13,4	10,7	8,6	6,9	5,5	4,4	3,6	2,8	2,2
315				28,6	23,2	18,7	15,0	12,1	9,7	7,7	6,2	4,9	3,9	3,2	2,5
355					26,1	21,1	16,9	13,6	10,9	8,7	7,0	5,5	4,4	3,5	2,8
400					29,4	23,7	19,1	15,3	12,3	9,8	7,9	6,3	5,0	4,0	3,2
450						26,7	21,5	17,2	13,8	11,0	8,8	7,0	5,6	4,5	3,6
500						29,6	23,9	19,1	15,3	12,3	9,8	7,8	6,2	5,0	4,0
560							26,7	21,4	17,2	13,7	11,0	8,8	7,0	5,6	4,4
630							30,0	24,1	19,3	15,4	12,3	9,8	7,9	6,3	5,0
710								27,2	21,8	17,4	13,9	11,1	8,8	7,1	5,6
800								30,6	24,5	19,6	15,7	12,5	10,0	7,9	6,3
900									27,6	22,0	17,6	14,0	11,2	8,9	7,1
1 000									30,6	24,5	19,6	16,6	12,4	9,9	7,9

Magnestams Reklam/Christensens Grafiska AB, Lerum 1989

PLASTIC PIPES FOR WATER SUPPLY AND SEWAGE DISPOSAL

by Lars-Eric Janson

Stockholm 1989

A growing interest in hydraulic pipeline conveyance of solids in recent years has resulted in a great many investigations into abrasion resistance in various pipe materials. For example refer to (55), (56) and (57), which all show a good verification of the findings presented above. Practical experience of using HDPE, MDPE and PVC pipes for transporting various industrial slurries also show that pipes made of 'lastics have a longer life in comparison with the steel pipes previously used.

The question of abrasion in plastic pipes has been the subject of particular concern in recent years in developing what have become known as "Light Weight Pipes" ; LWP). The design-concept of an LWP system implies that the two outer sheets of ; which the pipe wall is composed can be made very thin, compared to what is normal in solid-walled pipes. For instance, with regard to the need for pure ring stiffness, it may prove financially beneficial and technically feasible to let the inner pipe sheet be 1 mm or even less in the smallest pipe diameters. Consequently, the question will arise as to what then is the power limit in practical terms, for the thickness of the inner sheet of the wall, taking abrasion etc. into consideration.

As a basis for the development of, e.g., the ULTRA-RIB pipe system, several investigations were studied, of which (58), (59) and (60) are the most significant. Thus, the resistance of the inner wall surface due to sand carried in waste water has been studied in concrete pipes in ref (58) and for concrete, PVC and HDPE in ref (59). In (60) the wear on the inner wall surface in Ø 110 mm PVC, HDPE and ABS pipes caused by cleansing tools and high pressure washing with water has been studied.

A summary conclusion is that abrasion due to sand carried in a straight PVC or DPE pipeline can be estimated at less than 0.5 mm during an operational period 100 years. The notches caused by normal cleansing tools are also estimated to ɜ less than 0.5 mm in a straight pipe.

ased upon these findings, the Swedish KP approval requires an inner wall ickness of an LWP of not less than 1.0 mm at any single point. This applies to pipes ith a diameter less than or equal to 275 mm. (However, it is recommended that the p wall be thicker in bends and branches as these sections are subjected to eavier wear than straight pipes). For pipes with a diameter above 300 mm, the ninimum inner wall thickness is recommended as 2.0 mm and for pipes with a liameter above 500 mm, not less than 3.0 mm.

4. THE DESIGNING OF PLASTIC PIPES

4.1 Structural designing technique

Internal hydraulic pressure

Static load

All plastic pressure pipes are designed to withstand a specified internal hydraulic pressure. This is the nominal pressure, p, which indicates the working pressure to which the completed pipeline may be subjected in continuous operation.

When the pipe is subjected to internal pressure, a hoop tensile force N per unit of length is induced in the pipe wall in accordance with Figure 4.1.1 where

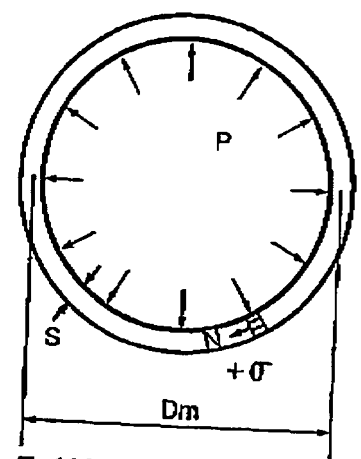

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Fig. 4.1.1
Pipe exposed to internal hydraulic pressure.

$$N = \frac{p\ D_m}{2} \qquad (4.1.1)$$

and where D_m is the mean diameter of the pipe, that is

$$D_m = \frac{(D+d)}{2} = D - s \qquad (4.1.2)$$

External diameter is denoted by D and the internal by d. If the tensile force is divided by the thickness of the wall, denoted by s, a hoop tensile stress will be obtained.

$$\sigma = \frac{p\ D_m}{2s} \qquad (4.1.3)$$

This tension is not allowed to exceed the permissible tensile stress during free creeping as described in Section 3.2. From this it may be seen that the permissible tensile stress, applied constantly for 50 years at a normal service temperature of 20°C, is 5 MPa for HDPE, 3.2 MPa for LDPE and 10 MPa for PVC according the current ISO standards. From the equation (4.1.3) the wall thickness s for a particular nominal pressure class (PN) corresponding to the working pressure p is defined as

$$s = \frac{p\ D_m}{2\sigma} \qquad (4.1.4)$$

In the ISO standard for thermoplastic pipes the external diameter D is constant irrespective of the pressure class. An expression of more practical use is obtained if D_m is replaced by the external pipe diameter of the pipe D. The expression for wall thickness then becomes

$$s = \frac{p\ D}{(2\sigma + p)} \qquad (4.1.5)$$

The most usual nominal pressure classes for PE are PN 2.5, PN 3.2, PN 4, PN 6 and PN 10 and for PVC PN 4, PN 6 and PN 10. These nominal pressures (PN) correspond to the respective maximum permissible working pressures, usually expressed in bars, as here.

By inserting the permissible values of tensile stress in equation (4.1.5) the following simple relationships between the wall thickness s and the standard working pressure PN is determined, (Table 4.1.1).

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Table 4.1.1

PN (bar)	s (mm if D I expressed in mm) HDPE	PVC
2.5	D/41	–
3.2	D/32.25	–
4	D/26	D/51
6	D/17.7	D/34.3
10	D/11	D/21

(The values in the denominator are used as a basis for standard classification in many countries. Cf Table 5.2.1)

Using the above calculation method summarised in the table, the wall thickness has been determined in most standards for thermoplastic pipes, which thus apply to a temperature of 20 °C and a duration of at least 50 years.

As is evident from Section 3.2, other permissible stress values than the above may be inserted in the equation (4.1.5) to determine the wall thickness of the pipe. A similar procedure may be employed when the operating temperature deviates from 20 °C, or if, for some reason, the chosen service life is other than 50 years. Figure 4.1.2 illustrates how the temperature affects the permissible internal hydraulic pressure for HDPE pipes of different pressure classes, designed according to the standard for the Type 1 grade. The corresponding diagrams for LDPE and PVC are shown in Figures 4.1.3 and 4.1.4 respectively.

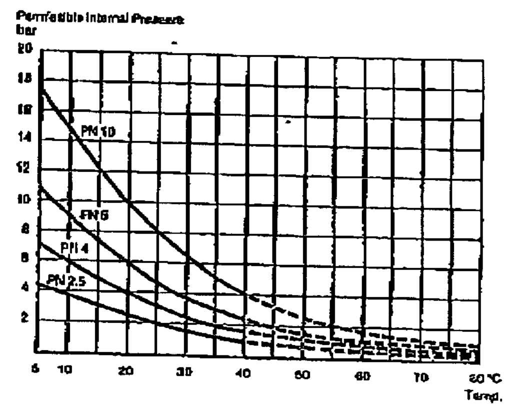

Fig. 4.1.2
Allowable internal hydraulic pressure as a function of temperature for HDPE, Type 1.

INTERNATIONAL STANDARD

ISO 1133

Second edition
1991-03-15

Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics

Plastiques — Détermination de l'indice de fluidité à chaud des thermoplastiques, en masse (MFR) et en volume (MVR)

Reference number
ISO 1133:1991(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 1133 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 1133:1981). This new edition includes, in addition to the previously described method for the determination of the melt mass-flow rate, a new procedure for the automatic measurement of both melt mass-flow rate and melt volume-flow rate.

Annex A forms an integral part of this International Standard.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics

1 Scope

1.1 This International Standard specifies a method for the determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastic materials under specified conditions of temperature and load. Normally, the test conditions for measurement of melt flow rate are specified in the material standard with a reference to this International Standard. The test conditions normally used for thermoplastics are listed in annex A. The melt volume-flow rate will normally be found useful when comparing filled and unfilled thermoplastics. The melt mass-flow rate can now be determined by automatic measurement provided the melt density at the test temperature is known.

1.2 The melt mass-flow rate and melt volume-flow rate of thermoplastics are dependent on the rate of shear. The rates of shear in this test are much smaller than those used under normal conditions of fabrication, and therefore data obtained by this method for various thermoplastics may not always correlate with their behaviour in actual use.

Both methods are useful in quality control.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1622-1:1985, *Plastics — Polystyrene (PS) moulding and extrusion materials — Part 1: Designation.*

ISO 1872-1:1986, *Plastics — Polyethylene (PE) and ethylene copolymer thermoplastics — Part 1: Designation.*

ISO 1873-1:1986, *Plastics — Polypropylene (PP) a propylene-copolymer thermoplastics — Part 1: Designation.*

ISO 2580-1:1990, *Plastics — Acrylonitril butadiene/styrene (ABS) moulding and extrusi materials — Part 1: Designation.*

ISO 2897-1:1990, *Plastics — Impact-resistant po styrene (SB) moulding and extrusion materials Part 1: Designation.*

ISO 4613-1:1985, *Plastics — Ethylene/vinyl aceta copolymer thermoplastics (E/VAC) — Part 1: Designation.*

ISO 4894-1:1990, *Plastics — Styrene/acrylonitr (SAN) copolymer moulding and extrusion materia — Part 1: Designation.*

ISO 6402-1:1990, *Plastics — Impact-resista acrylonitrile/styrene moulding and extrusion m terials (ASA, AES, ACS), excluding butadien modified materials — Part 1: Designation.*

ISO 6507-1:1982, *Metallic materials — Hardness te — Vickers test — Part 1: HV 5 to HV 100.*

ISO 7391-1:1987, *Plastics — Polycarbonate mouldi and extrusion materials — Part 1: Designation.*

ISO 7792-2:1988, *Plastics — Polyalkylene terephtha ates — Part 2: Preparation of test specimens a determination of properties.*

ISO 8257-1:1987, *Plastics — Poly(methyl methac late) (PMMA) moulding and extrusion materials Part 1: Designation.*

ISO 9988-1:1991, *Plastics — Polyoxymethylene (PO moulding and extrusion materials — Part 1: Desi nation.*

3 Apparatus

3.1 Basic apparatus

The apparatus is basically an extrusion plastometer (capillary rheometer) operating at a fixed temperature. The general design is as shown in figure 1. The thermoplastic material, which is contained in a vertical metal cylinder, is extruded through a die by a loaded piston. The apparatus consists of the following essential parts:

3.1.1 Steel cylinder, fixed in a vertical position and suitably insulated for operation up to 400 °C. The cylinder length shall be between 115 mm and 180 mm and the internal diameter 9,55 mm ± 0,025 mm. The base of the cylinder shall be thermally insulated in such a way that the area of the exposed metal is less than 4 cm^2, and it is recommended that an insulating material such as Al_2O_3 ceramic fibre or another suitable material be used in order to avoid sticking of the extrudate.

The bore shall be suitably hardened to a Vickers hardness of no less than 500 (HV 5 to HV 100) (see ISO 6507-1). A piston guide shall be provided to prevent additional friction caused by misalignment of the piston.

3.1.2 Steel piston, having a working length at least as long as the cylinder. The piston shall have a head 6,35 mm ± 0,1 mm in length. The diameter of the head shall be less than the internal diameter of the cylinder by 0,075 mm ± 0,01 mm. The lower edge of the head shall have a radius of 0,4 mm and the upper edge shall have its sharp edge removed. Above the head, the piston shall be relieved to about 9 mm diameter. A stud may be added at the top of the piston to support the removable load, but the piston shall be thermally insulated from the load. Along the piston stem, two thin annular reference marks shall be scribed 30 mm apart and so positioned that the upper one is aligned with the top of the cylinder when the distance between the lower edge of the piston head and the top of the die is 20 mm. These annular marks on the piston are used as reference points during the determination (see 6.3 and 7.4.3).

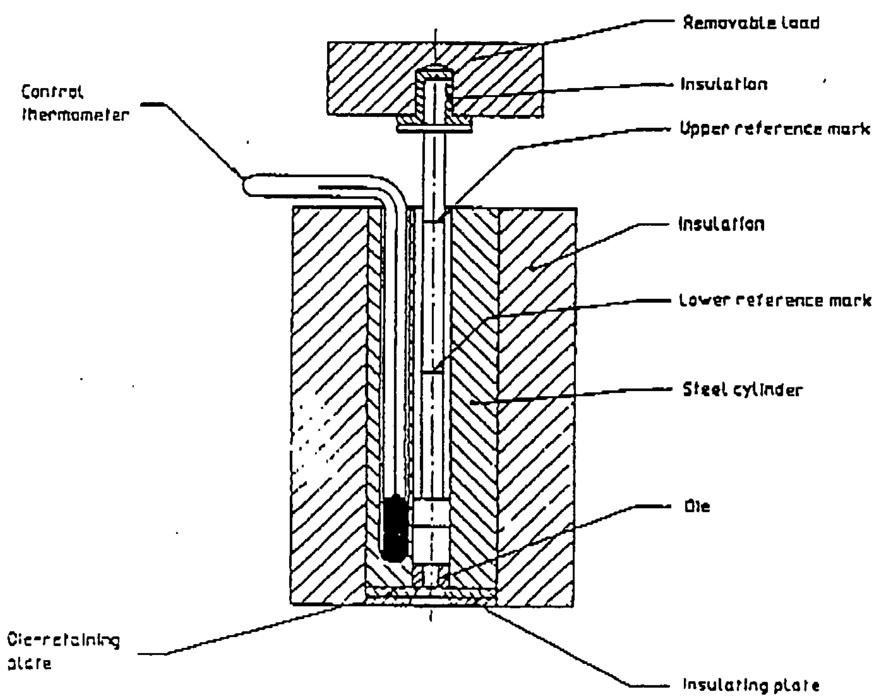

Figure 1 — Typical apparatus for determining melt flow rate (showing one of the possible methods of retaining the die and one type of piston)

To ensure satisfactory operation of the apparatus, the cylinder and the piston shall be made of steel of different hardness. It is convenient for ease of maintenance and renewal to make the cylinder of the harder steel.

The piston may be either hollow or solid. In tests with lower loads, the piston shall be hollow, otherwise it may not be possible to obtain the lowest prescribed load. When the test is performed with the higher loads, the hollow piston is not desirable, as the higher load may distort such a piston. In such tests, a solid piston or a hollow piston with suitable guides shall be used. When using this latter modification, it is essential that the heat loss along the piston, which is generally longer than usual, does not alter the test temperature of the material.

3.1.3 **Temperature-control system**, such that the selected temperature of the material in the cylinder can be maintained to within ± 0.5 °C. Automatic temperature control is strongly recommended.

3.1.4 **Dies**, made of tungsten carbide or hardened steel, 8.000 mm ± 0.025 mm in length. The interior shall be circular, straight and uniform in diameter such that in all positions it is within ± 0.005 mm of a true cylinder of nominal diameter 2.095 mm.

The bore shall be suitably hardened to a Vickers hardness of no less than 500 (HV 5 to HV 100) (see ISO 6507-1). The die shall not project beyond the base of the cylinder (see figure 1) and shall be mounted so that its bore is co-axial with the cylinder bore.

3.1.5 **Means of setting and maintaining the cylinder truly vertical.**

A two-directional bubble level, set normal to the cylinder axis, and adjustable supports for the apparatus are suitable for the purpose. This is to avoid excessive friction caused by the piston or bending under heavy loads.

3.1.6 **Removable load**, on the top of the piston, which consists of a set of weights which may be adjusted so that the combined mass of the load and the piston gives the selected nominal load to an accuracy of ± 0.5 %. An alternative mechanical loading device may be used for the higher loads.

3.2 Accessory equipment

3.2.1 General

3.2.1.1 **Equipment for introducing samples into the cylinder**, consisting of a packing rod made of non-abrasive material.

3.2.1.2 **Cleaning equipment.**

3.2.1.3 **Mercury-in-glass thermometer** (calibrati thermometer) or another temperature-measuri device. This measuring device shall be calibrated permit temperature measurement to ± 0.1 °C at t temperature and immersion conditions to be us when calibrating the temperature-control system accordance with 5.1.

3.2.2 For procedure A

3.2.2.1 **Cutting tool**, for cutting off the extrude sample. A sharp-edged spatula has been fou suitable.

3.2.2.2 **Stop-watch**, accurate to ± 0.1 s.

3.2.2.3 **Balance**, accurate to ± 0.5 mg.

3.2.3 For procedure B

Measurement equipment, for the automat measurement of distance and time for the pistc movement.

The equipment shall have the capacity to obta three measurements for each sample in the cyli der.

4 Test specimen

4.1 The test specimen may be in any form that ca be introduced into the bore of the cylinder, for e ample powder, granules or strips of films.

NOTE 1 Some materials in powder form do not give bubble-free filament if they are not previously pressed.

4.2 The test specimen shall be conditioned and, necessary, stabilized prior to the test, in accordanc with the material specifications.

5 Temperature calibration, cleaning and maintenance of the apparatus

5.1 Calibration of the temperature-control system

5.1.1 Verify the accuracy of the temperature-contrc system (3.1.3) at least once each day that the appa ratus is used or whenever the temperature of test i changed, whichever is the more frequent. For thi purpose, adjust the cylinder temperature-contrc system until the cylinder will remain at the require temperature as indicated by the control ther mometer. Preheat a calibration thermomete (3.2.1.3) to the same temperature. Then charge th cylinder with a small quantity (3 to 4 pellets) of th material to be tested, or a material representativ thereof (see 5.1.2), using the same technique as fc a test (see 6.2). Four minutes after completing th

charging of the material, introduce the calibration thermometer into the sample chamber and immerse it in the material therein until the tip of the bulb is 10 mm from the upper face of the die. After a further interval of at least 4 min, correct the temperature indicated by the control thermometer by algebraic addition of the difference between the temperatures read on the two thermometers.

5.1.2 It is essential that the material used during calibration be sufficiently fluid to permit, for instance, a mercury-filled thermometer bulb to be introduced without excessive force and risk of damage. A material with an MFR of greater than 45 g/10 min (2,16 kg charge) at the temperature of calibration has been found suitable.

If such a material is used for calibration purposes in place of a more viscous material which is to be tested, the dummy material shall have a thermal diffusivity similar to that of the material to be tested, so that warm-up behaviour is similar. It is necessary that the quantity charged for calibration be such that, when the calibration thermometer is subsequently introduced, an appropriate portion of the thermometer is immersed for accurate temperature measurement. This can be checked by inspecting the level of material coating the end of the calibration thermometer, removing the thermometer from the cylinder if necessary.

5.2 Cleaning the apparatus

The apparatus shall be cleaned thoroughly after each determination. The cylinder may be cleaned with cloth patches. The piston shall be cleaned while hot with a cloth. The die may be cleaned with a closely fitting brass reamer or wooden peg. Pyrolytic cleaning in a nitrogen atmosphere at about 550 °C may also be used. On no account shall abrasives or materials likely to damage the surface of the piston, cylinder or die be used.

5.3 Maintenance of apparatus

It is recommended that, at fairly frequent intervals, for example once a week for instruments in constant use, the insulating plate and the die-retaining plate, if fitted as in figure 1, be removed, and the cylinder cleaned throughout.

6 Procedure A

6.1 Clean the apparatus (see 5.2). Before beginning a series of tests, ensure that the cylinder (3.1.1) and piston (3.1.2) have been at the selected temperature for not less than 15 min.

6.2 Then charge the cylinder with 3 g to 8 g of the sample according to the anticipated melt flow rate (see, for example, table 1). During the charging, compress the material with the packing rod (3.2.1.1), using hand pressure. To ensure a charge as free from air as possible for material susceptible to oxidative degradation, complete the charging process in 1 min. Put the piston, loaded or unloaded according to the flow rate of the material, in the cylinder.

If the melt flow rate of the material is high, that is, more than 10 g/10 min, the loss of sample during preheating will be appreciable. In this case, use an unloaded piston or one carrying a smaller weight during the preheating period, and then change to the desired weight at the end of the 4 min preheating time.

Table 1

Melt flow rate g/10 min	Mass of test portion in cylinder[1)] g	Extrudate cut-off time-interval s
0,1 to 0,5	3 to 5	240[2)]
> 0,5 to 1	4 to 5	120
> 1 to 3,5	4 to 5	60
> 3,5 to 10	6 to 8	30
> 10	6 to 8	5 to 15[3)]

1) When the density of the material is greater than 1,0 g/cm^3, it may be necessary to increase the mass of the test portion.

2) It is recommended that melt flow rate should not be measured if the value obtained in this test is less than 0,1 g/10 min or greater then 100 g/10 min.

3) To achieve adequate repeatability when testing materials having an MFR greater than 25 g/10 min, it may be necessary either to control and measure cut-off intervals automatically to less than 0,1 s or to use procedure B.

6.3 Four minutes after completing the introduction of the test portion, during which time the temperature shall have returned to that selected, place the selected load on the piston, if it was unloaded or under-loaded. Depending on the actual viscosity of the material, allow the piston to descend under gravity or push it down faster using hand pressure, until a bubble-free filament is extruded. The time for this operation shall not exceed 1 min. Cut off the extrudate with the cutting tool (3.2.2.1), and discard. Then allow the loaded piston to descend under gravity. When the lower reference mark has reached the top edge of the cylinder, start the stopwatch (3.2.2.2), and simultaneously cut off the extruded portion with the cutting tool and again discard.

Then collect successive cut-offs in order to measure the extrusion rate, at time-intervals, depending on the melt flow rate, so chosen that the length of a single cut-off is not less than 10 mm and preferably between 10 mm and 20 mm (see cut-off time-intervals in table 1 as a guide).

For low values of MFR (and MVR), it may not be possible to take a cut-off with a length of 10 mm or more within the maximum time-interval of 240 s. In this case, procedure B shall be used.

Stop cutting when the upper mark on the piston stem reaches the top edge of the cylinder. Discard any cut-off containing visible air bubbles. After cooling, weigh individually, to the nearest 1 mg, the remaining cut-offs, which shall number at least three, and calculate their average mass. If the difference between the maximum and the minimum value of the individual weighings exceeds 15 % of the average, discard the result and repeat the test on a fresh portion of the sample.

The time between charging the cylinder and the last measurement shall not exceed 25 min.

6.4 The melt mass-flow rate (MFR), expressed in grams per 10 min, is given by the equation

$$\mathrm{MFR}(\theta, m_{\mathrm{nom}}) = \frac{t_{\mathrm{ref}}\, m}{t}$$

where

- θ is the test temperature, in degrees Celsius;
- m_{nom} is the nominal load, in kilograms;
- m is the average mass, in grams, of the cut-offs;
- t_{ref} is the reference time (10 min), in seconds (600 s);
- t is the cut-off time-interval, in seconds.

Express the result to two significant figures.

7 Procedure B

7.1 Principle

The melt mass-flow rate (MFR) and the me volume-flow rate (MVR) are determined by using e ther of the following two principles:

a) measurement of the distance the piston move in a specified time;

or

b) measurement of the time in which the pisto moves a specified distance.

7.2 Optimum measurement accuracy

For repeatable determination of MFR betwee 0,1 g/10 min and 50 g/10 min or MVR betwee 0,1 cm³/10 min and 50 cm³/10 min, the movemer of the piston has to be measured to the neares ± 0,1 mm and the time to an accuracy of 0,1 s.

7.3 Pretreatment

Follow procedure A specified in 6.1 to 6.3 (to end c first paragraph).

7.4 Determination

7.4.1 When the lower reference mark has reache the top edge of the cylinder, start the automati measurement. Proceed as specified in 7.4.2 a) if us ing the principle given in 7.1 a) or as specified i 7.4.2 b) if using the principle given in 7.1 b).

7.4.2 Measure

a) the distance moved by the piston at predete mined times (three or more)

or

b) the times taken by the reference mark to cove a specified distance (three or more).

Stop the measurement when the upper mark on th piston stem reaches the top edge of the cylinder.

7.4.3 The time between charging the cylinder an the last measurement shall not exceed 25 min

7.5 Expression of results

7.5.1 The melt volume-flow rate (MVR), expressed in cubic centimetres per 10 min, is given by the equation

$$\mathrm{MVR}(\theta, m_{nom}) = \frac{A \cdot t_{ref} \cdot l}{t} = \frac{427 l}{t}$$

where

θ is the test temperature, in degrees Celsius;

m_{nom} is the nominal load, in kilograms;

A is the mean cross-sectional area, in square centimetres, of the piston and the cylinder (= 0,711 cm²);

t_{ref} is the reference time (10 min), in seconds (600 s);

t is the predetermined time of measurement [see 7.4.2 a)] or the mean value of individual time measurements [see 7.4.2 b)], in seconds;

l is the predetermined distance moved by the piston [see 7.4.2 b)] or the mean value of individual distance measurements [see 7.4.2 a)], in centimetres.

7.5.2 The melt mass-flow rate (MFR), expressed in grams per 10 min, is given by the equation

$$\mathrm{MFR}(\theta, m_{nom}) = \frac{A \cdot l \cdot \rho \cdot t_{ref}}{t} = \frac{427 l \cdot \rho}{t}$$

where

θ, m_{nom}, A, l, t_{ref} and t are as defined in 7.5.1;

ρ is the density, in grams per cubic centimetre, of the melt at the test temperature and is given by the equation

$$\rho = \frac{m}{0{,}711 l}$$

m being the mass, determined by weighing, of a known extruded volume of length l.

7.5.3 Express the result to two significant figures.

8 Precision

When the method is used with certain materials, consideration shall be given to the factors leading to a decrease in repeatability. Such factors include

a) thermal degradation or crosslinking of the material, causing the melt flow rate to change during the preheating or test period; powdered materials requiring long preheating times are sensitive to this effect and, in certain cases, the inclusion of stabilizers is necessary to reduce the variability;

b) filled or reinforced materials, where the distribution or orientation of the filler may affect the melt flow rate.

The precision of the method is not known because inter-laboratory data are not available. A single precision statement would not be suitable because of the number of materials covered. However, a coefficient of variation of about ± 10 % could be expected.

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the test sample, including the physical form of the material with which the cylinder was charged;

c) the details of conditioning;

d) the details of any stabilization (see 4.2);

e) the temperature and load used in the test;

f) the melt mass-flow rate, in grams per 10 min, or the melt volume-flow rate, in cubic centimetres per 10 min, expressed to two significant figures;

g) a report of any unusual behaviour of the test portion, such as discoloration, sticking, extrudate distortion or unexpected variation in melt flow rate.

Annex A
(normative)

Test conditions for melt flow rate determination

The conditions used shall be as indicated in the appropriate material designation or specification. Table A.1 indicates test conditions that have been found useful. Table A.2 indicates test conditions that are presently specified in relevant International Standards. Other test conditions not listed here may be used, if necessary, for a particular material.

Table A.1

Conditions No.	Conditions Code-letter	Test temperature, θ °C	Nominal load (combined), m_{nom} kg
1	A	250	2,160
2	B	150	2,160
4	D	190	2,160
6	F	190	10,000
7	G	190	21,600
8	H	200	5,000
12	M	230	2,160
13	N	230	3,800
17	S	280	2,160
18	T	190	5,000
19	U	220	10,000
21	W	300	1,200
22	Z	125	0,325

NOTE — If, in the future, conditions other than those listed in this table are necessary, e.g. for new thermoplastics, only the loads already in use shall be chosen. Temperatures shall also be selected from those already in the table. If absolutely necessary, new temperatures might have to be taken because of the nature of the new thermoplastic. In this case, application to ISO/TC 61/SC 5 shall be made to include the new conditions. If approved, a suitable code-letter will provisionally be issued and the standard amended at the 5-year revision.

Table A.2

International Standard (see clause 2)	Materials	Conditions No.	Conditions Code-letter	Test temperature, θ °C	Nominal load (combined), m_{nom} kg
ISO 1622-1	PS	8	H	200	5,000
ISO 1872-1 ISO 1872-1 ISO 1872-1	PE PE PE	4 7 18	D G T	190 190 190	2,160 21,600 5,000
ISO 1873-1	PP	12	M	230	2.160
ISO 2580-1	ABS	19	U	220	10,000
ISO 2897-1	Impact-resistant PS	8	H	200	5,000
ISO 4613-1 ISO 4613-1 ISO 4613-1	E/VAC E/VAC E/VAC	2 4 22	B D Z	150 190 125	2,160 2,160 0,325
ISO 4894-1	SAN	19	U	220	10.000
ISO 6402-1	ASA	19	U	220	10,000
ISO 7391-1	PC	21	W	300	1,200
ISO 7792-2 ISO 7792-2	PET PBT	17 1	S A	280 250	2.160 2,160
ISO 8257-1	PMMA	13	N	230	3,800
1) 1)	PB PB	4 6	D F	190 190	2,160 10,000
ISO 9988-1	POM	4	D	190	2.160
1)	MM/ABS	19	U	220	10.000

1) No designation standard available yet.